Nitrogen sensitivities of a sample of commercial hot cathode ionization gage tubes

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In order to help assess the magnitude of errors that might arise from the use of uncalibrated ionization gage tubes and help select the best type of tube when accurate measurements are required we have determined the nitrogen sensitivities from 10^{-5} to 10^{-2} Pa for lots of from two to four each of five different types of commerical hot cathode gage tubes. Included were conventional triodes and B-A structures of four distinct types. Two types with tungsten filaments—the triodes and tubulated B-A structures—were markedly superior to the others with respect to agreement with manufacturer's sensitivity, linearity, and uniformity of sensitivity within a type. The least satisfactory results were obtained from the nude B-A structures, which showed significant nonlinearities, sensitivities ranging from 70%–110% of the manufacturer's value, and typical 25% differences in sensitivity between the two filaments of a dual-filament structure.

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I. INTRODUCTION

A growing number of industrial processes and research and development projects require accurate pressure measurements in the high vacuum range. Ionization gages are used for the vast majority of measurements in this range, with the hot cathode or hot filament type generally regarded as the most stable. In order to help assess the magnitude of errors that might arise from the use of uncalibrated gage tubes and help select the best type of tube when accurate measurements are required the authors have determined the nitrogen sensitivities from 10^{-5} to 10^{-2} Pa for lots of from two to four each of five different types of commercial gage tubes.

The criteria on which these tubes are evaluated include agreement with manufacturer's stated sensitivity, linearity, and uniformity of sensitivity within a type. These tubes are identified by generic type, but as a matter of policy, not by manufacturer or model. Before final selections can be made, reproducibility, long-term stability, and freedom from sensitivity changes in shipment will have to be evaluated. The measurements reported in this paper were performed by a method that is appropriate for calibrating gage tubes to be employed when pressure measurements with uncertainties of ±10% are acceptable in the high vacuum range.

II. DEFINITIONS AND TERMS

(1) *Ionization gage tube.* This term is used in the present paper in a broad sense to encompass all types of hot cathode ionization gage tubes, including nude structures.

(2) *Sensitivity.* Let i_+ represent the ion collector current of an ionization gage tube operating at constant emission current i_- and pressure P. Then if i_0 is the value of i_+ at the base pressure P_0 of the vacuum system, the sensitivity S as employed in the present paper is defined by the equation

$$S = \frac{i_+ - i_0}{i_-(P - P_0)}.$$

Note that this definition differs from others that do not subtract residual current from the total collector current.

III. APPARATUS AND PROCEDURES

The sensitivities of the tubes under test were determined by direct comparison with reference gage tubes on a continuous-flow comparator. Calibrated reference gage tubes were obtained from the National Physical Laboratory, Teddington, Great Britain. The stainless steel vacuum chamber was in the form of an upright cylinder with an inside diameter of 0.30 m and a height of 0.63 m. Nitrogen was admitted to the chamber from a reservoir through an adjustable leak valve. The upward-directed gas inlet tube, with its exit 0.5 m above the level of the gage ports, was coaxial with the chamber within 5 mm. An ion pump with a rated nitrogen speed of 270 l s^{-1} pumped the chamber continuously from below. A plate with a circular orifice centered on the axis could be lowered into contact with the bottom of the chamber to throttle the volume rate of flow to a nominal 10 l s^{-1}, or raised to achieve a higher effective pumping speed during evacuation and bakeout.

The chamber had eight ports equally spaced on a horizontal circle 6 cm above the bottom. Of these parts, one served as a roughing port, one admitted the gas inlet tube, and a third introduced a bellows-sealed lever for manipulating the orifice plate. The remaining five ports were available for attaching gage tubes. Each port had a calculated conductance of 55 l s^{-1} for nitrogen. All flanges were sealed with copper gaskets. A cylindrical aluminum baffle 0.2 m in diameter was attached to the orifice plate to serve the dual purpose of preventing line-of-sight communication between tubulated gages (not, however, between nude gages) and of reducing any pressure differences between gage ports.

The gage tubes under investigation included one type of conventional triode with its cylindrical collector surrounding the helical grid and central filament, and four distinct Bayard–Alpert (B–A) types: a tubulated type of standard size with glass envelope and single filament of thoriated iridium, a tubulated type of standard size with glass envelope and dual tungsten filaments, a dual-filament miniature tube with one tungsten filament and the other of thoriated iridium, and a nude type with dual tungsten filaments. For most types a three-tube lot of identical origin was tested by concurrent comparison with a reference gage. However, only two B–A gage tubes with dual tungsten filaments were tested, but four B–A tubes with single thoriated iridium filament were included in the study. All tubes or nude structures were mounted on metal-gasketed flanges. The nude structures were supplied in this form by the manufacturer. For all other types the method of mounting preserved the tubulation conductance of the unit as supplied by the manufacturer.

All gage tubes were operated on commercial controllers, with added external circuitry or internal rewiring as necessary to supply the interelectrode potentials and stabilize the emission currents at the values specified by the tube manufacturers. For all cases in which the manufacturers did not specify the emission current, the tests were conducted with this current controlled at 1 mA. For the reference gage the emission current was measured at the filament, but for those under test it was obtained from the potential difference across a measured resistance in the grid return. All voltages were read with a digital voltmeter and ion collector currents were measured with a calibrated digital picoammeter.

Before a set of gages was tested the system was evacuated and baked for approximately 24 h at a maximum temperature of 230°C. The gage tubes were further degassed while the system cooled, after which the gages were operated for at least 15 h at the voltages and currents specified for the test before nitrogen was admitted. All gage types except the conventional triodes were degassed by electron bombardment. The latter type was degassed by ohmic heating of the grid. With the orifice plate in the raised (open) position the typical base pressure in the system following this treatment gave an indication of less than 3×10^{-7} Pa (2.2×10^{-9} Torr) on a Bayard–Alpert gage calibrated for nitrogen. This pressure indication increased approximately 10% when the plate was lowered. The ion current due to residual gas was approximately 1% of the total ion current at the lowest nitrogen pressure at which sensitivities were determined. To the extent that the ratios of sensitivity for residual gas to sensitivity for nitrogen are comparable for different gages, effects due to residual gas tend to cancel when sensitivities are determined by direct comparison with a calibrated ionization gage. Hence, estimated uncertainties due to residual gas are well below the 1% level.

With the system at base vacuum the emission current and ion collector current indications were recorded for each gage. The orifice plate was then lowered and the test was carried out. Pressures were established in ascending order. The system was allowed to stabilize at each leak setting for at least 20 min before the gage indications were recorded. In order to correct for slow pressure drifts, typically steady and smaller in magnitude than 0.1% per min, which are ascribed to variations in the conductance of the leak valve, each gage to be tested was read twice at times bracketing the reference gage reading, and these indications were interpolated to the time of the reference reading.

IV. UNCERTAINTIES

Sensitivities obtained by this method are reported with an estimated maximum relative uncertainty (90% confidence limits) of ±5%. This overall uncertainty includes a ±2% uncertainty in the reference gage tube sensitivity, a ±1.5% uncertainty ascribed to measurement and control of the electrical parameters of the reference gage, and a similar ±1.5% due to electrical measurements on the gage under test. As noted above, estimated uncertainties due to residual gas are too small to contribute significantly to the total. An ionization gage of the type employed in a recent intercomparison of European vacuum standards[1] remained on the system throughout most of the present investigation. It provided data consistent with the hypothesis that the reference gage sensitivity remained reproducible within the stated limits of uncertainty. Direct comparison of a spinning rotor gage with the reference ionization gage gave a value for the coefficient of tangential momentum transfer between nitrogen molecules and the steel rotor agreeing to within ±1% with recently published values.[2]

V. RESULTS AND DISCUSSION

The sensitivities obtained from these measurements are plotted vs pressure in Figs. 1–4. (Note that 1 Torr = 1.33 Pa) In all figures the indicated ranges of sensitivity values are with respect to the manufacturers' stated sensitivity. Of the B–A tubes, the tubulated type with dual tungsten filaments exhibited the best linearity and sensitivities in closest agreement with the value quoted by the manufacturer. As Fig. 1 indicates, both filaments of gage tube number 4 and one filament of tube number 1 gave sensitivities agreeing with each other within ±1%.

All other B–A types showed much larger deviations from the sensitivities quoted by the manufacturers, with a notice-

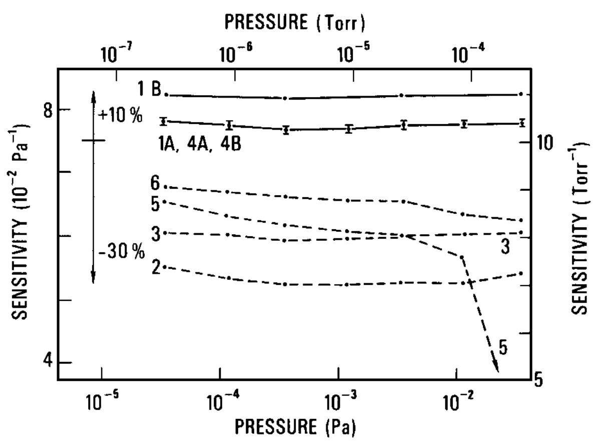

FIG. 1. Nitrogen sensitivities of six tubulated B-A tubes of standard size. Dashed lines: thoriated iridium filaments. Solids lines: tungsten filaments. A and B distinguish the two filaments on each of gage tubes 1 and 4. Vertical arrows indicate the range of deviations from the manufacturer's stated sensitivity (horizontal line).

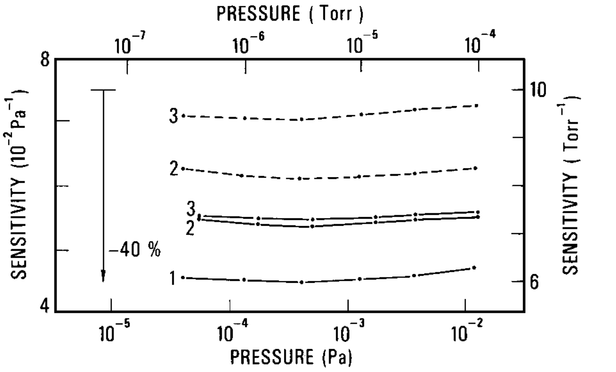

FIG. 2. Nitrogen sensitivities of three miniature B-A gage tubes with dual filaments. The numbers 1–3 identify the tubes. Dashed lines: thoriated iridium filaments. Solid lines: tungsten filaments. The vertical arrow indicates the range of deviations from the manufacturer's stated sensitivity (horizontal line). The thoriated filament of tube 1 was not calibrated.

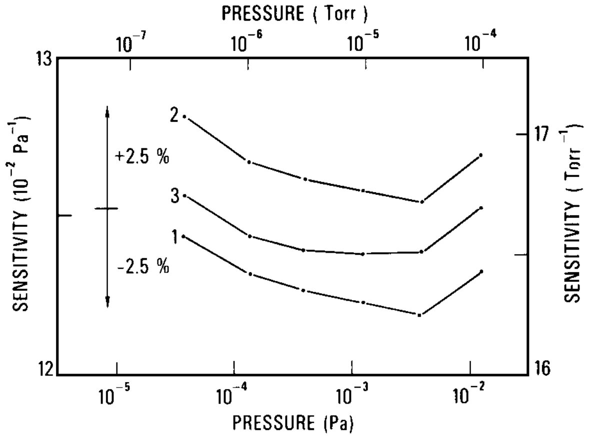

FIG. 4. Nitrogen sensitivities of three conventional triode gage tubes, identified by the numbers 1–3. Vertical arrows indicate the range of deviation from the manufacturer's stated sensitivity (horizontal line). The evident systematic nonlinearities are not meaningful since they are less than the uncertainty of the reference gage readings.

able trend towards sensitivities below the stated values. The miniature B–A tubes gave sensitivities as much as 40% low, and the tubulated type with thoriated iridium filaments showed sensitivities as much as 30% low and a marked nonlinearity in one case.

Sensitivities for the nude B–A structures ranged from 10% above to 30% below the value quoted in the manufacturer's literature, with significant nonlinearity evident in all cases. In Fig. 3 the filaments of each nude structure are consistently designated A and B on the basis of their location with respect to the other electrodes. Filament B gave sensitivity values typically 25% greater than A in all three cases. This result might be due to the unsymmetrical location of the filaments with respect to the supporting structure in this particular model. These observations indicate that the two filaments of an uncalibrated nude structure of this particular type are not interchangeable in any critical application. In addition, when nude structure number 2 operating on filament A was housed in a grounded stainless steel tube with an inside diameter of 3.5, cm, the sensitivity at 1.3×10^{-4} Pa was 15% greater than when it was mounted nude in the gage port. This and similar observations from fragmentary tests not included in Fig. 3 indicate that the sensitivity of a nude B–A structure can be significantly affected by the electrical boundary conditions under which it is operated. In apparent contrast with the latter conclusion, however, is the observation that the sensitivity of a nude gage, operating in close proximity to and in line-of-sight communication with another of the same type, was essentially unaffected by its neighbor. This observation was made by following the sensitivity of one nude structure at 1.3×10^{-2} Pa and noting that this value remained constant within 0.1% when its neighbor, less than 10 cm away, was turned off. This result indicates that the sensitivities reported for these nude structures are not invalidated by gage interactions.

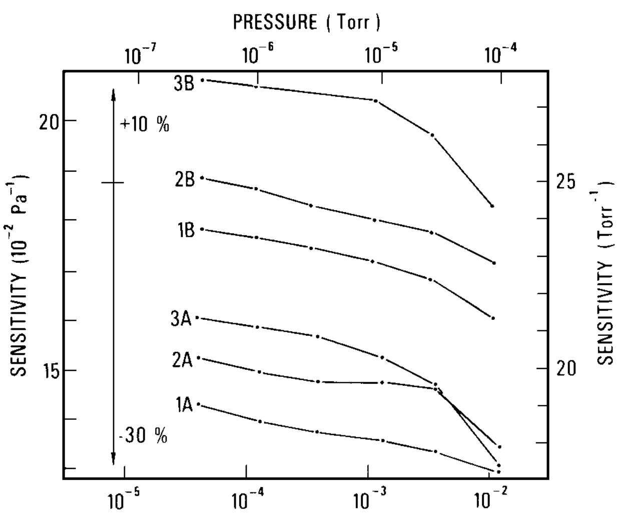

FIG. 3. Nitrogen sensitivities of three nude B-A structures with dual tungsten filaments. The numbers 1–3 identify the structures; the letters A and B the filaments. Vertical arrows indicate the range of deviations from the manufacturer's stated sensitivity (horizontal line).

Of all the gage tubes tested, the conventional triodes exhibited the closest consistency within the sample lot and with the sensitivity quoted by the manufacturer. The average deviation from this rated sensitivity is less than 2%, well within the estimated uncertainty of ±5%. It would be worthwhile to evaluate the long-term stability of these triode tubes and the tubulated B–A structures with dual tungsten filaments.

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